

ULTRASONIC FREQUENCY SELECTION FOR AQUEOUS FINE CLEANING

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Abstract

A study was conducted to evaluate ultrasonic cleaning systems for precision cleaning effectiveness for oxygen service hardware. This evaluation was specific for Rocketdyne Div. of Rockwell Aerospace alloys and machining soils. Machining lubricants and hydraulic fluid were applied as soils to standardized complex test specimens designed to simulate typical hardware. The study consisted of tests which included 20, 25, 30, 40, 50, and 65 kHz ultrasonic cleaning systems. Two size categories of cleaning systems were evaluated, 3- to 10-gal laboratory size tanks and 35- to 320-gal industrial size tanks. The system properties of cavitation; frequency vs. cleaning effectiveness; the two types of transducers; and the power level of the system vs. size of the cleaning tank were investigated. The data obtained from this study was used to select the ultrasonic tanks for the aqueous fine clean facility installed at Rocketdyne.

INTRODUCTION

The technique to preclean and fine clean Rocketdyne hardware includes the use of 1,1,1-trichloroethane (TCA), an ozone depleting chemical that will be banned as of 12/31/95. Rocketdyne is committed to using aqueous cleaning techniques to replace the majority of the cleaning processes in order to minimize environmental impact.

The use of TCA provides a very effective cleaning which is achieved mainly through chemical action and relies very little on mechanical action. To change to aqueous cleaning techniques requires that the majority of the cleaning be achieved through mechanical action as the effectiveness of the chemical action is greatly reduced with aqueous cleaning agents. The mechanical technique selected for the fine cleaning process was ultrasonic cavitation. The selection of ultrasonic cleaning for aqueous fine cleaning was influenced by the work performed by IBM General Products Division, San Jose, CA;[1,2] Aerojet, Sacramento, CA; Newark Air Force Base, Ohio;[3] and NASA Kennedy Space Center, Florida.[4]

A study was conducted to select the most effective ultrasonic cleaning system for the fine cleaning requirements at Rocketdyne. Each manufacturing environment is unique for the hardware alloys and soils generated. From the results of previous studies, it was determined that the ultrasonic fine cleaning process was to include a three-step cleaning process. In addition, this study evaluated the possibility of enhanced cleaning capability by varying the frequency within the steps of the cleaning process. The theory reported by D. H. McQueen suggested that cleaning with low ultrasonic frequencies were more effective in removing large deposits of contamination and higher frequencies were more effective in removing microlayers of hydrocarbons.[5]

For the purpose of this study, low ultrasonic frequency is defined as 20, 25, and 30 kHz and high frequency is defined as 40, 50, and 65 kHz. This range of frequencies was limited due to the availability of equipment for industrial size cleaning systems. The equipment manufacturers offer cleaning systems which differ in frequencies, types of transducers and levels of power. The selection of equipment for the Rocketdyne facility was based on comparing the cleaning effectiveness of system combinations that were available at the time of this study.

BACKGROUND

There were several properties associated with ultrasonic cleaning that Rocketdyne found necessary to learn in detail in order to effectively discuss the various aspects of the ultrasonic cleaning

equipment. These properties were the phenomenon of cavitation, the frequencies available, the types of transducers, and the power levels required.

Ultrasonic Cavitation

Cavitation is the mechanism by which ultrasonic tank systems clean. Cavitation is defined as the formation and collapse of vapor bubbles in liquids by means of a mechanical force. In ultrasonic cleaning, the mechanical force is the sound wave. Sound waves are longitudinal mechanical waves that can be transmitted through solids, liquids, or gases. There is a large range of frequencies in which these waves can be generated. The human hearing range is from about 20 cycles/sec to about 20,000 cycles/sec. Ultrasonic waves are sound waves above 16,000 cycles/sec (16 kHz).[6,7]

A sound wave is transmitted through a liquid producing a series of compressions and rarefactions. The compression zone in a half-cycle of the sound wave exerts a positive pressure on the molecules of the liquid and pushes them together. The rarefaction zone in the other half-cycle of the sound wave exerts a negative pressure which pulls the molecules away from each other.[6] The force in this latter half-cycle will form a cavity when the negative pressure generated is great enough to overcome the surface tension (tensile strength) of the liquid. The ideal cavity is the generation of a vacuum bubble. In the negative pressure zone of the sound wave, a drop in pressure reduces the boiling point of the liquid creating a vapor bubble. This vapor recondenses to liquid as a result of an increase in pressure due to the positive pressure zone created in the other half-cycle of the sound wave creating a vacuum bubble. When the bubble grows large enough, it implodes with great violence producing liquid jets and shock waves, minute areas of extremely high temperature (about 5000K) and very high pressures.[7,8]

The size of the cavitation bubble is dependent on the frequency of the sound wave, the higher the frequency, the smaller the size of the bubble. The relationship of the size of the cavitation bubble to frequency under normal atmospheric pressure was roughly given by D. H. McQueen:

$$R_0 = 300/f,$$

where R_0 is the equilibrium bubble radius in cm and f is the frequency in Hz.[5] This relationship is a simplified version of the derivation presented by Noltingk and Neppiras for the relationships for cavitation bubble dynamics.[9] For an ultrasonic cleaning system that is 20 kHz, the cavitation bubble size is about 150 μm ; for 40 kHz, the bubble size is about 75 μm . The lower frequencies produce larger cavitation bubbles which have a greater implosion energy, as it requires more work to generate the larger bubble.[10] Neppiras has reported that not only does the cavitation intensity increase with reduced frequency, but also the cavitation threshold is decreased.[11]

The volume concentration of the cavitation bubbles can be increased by increasing the amplitude or intensity of the sound wave until the density of bubbles reaches a self-limiting state. This state is known as unloading. This condition is a result of the fact that sound is transmitted from one medium to another only when the densities of the two media are similar. The density difference between air and water is too great for the transmission of sound from one to the other. Therefore, if a layer of cavitation bubbles becomes too concentrated at the surface of the radiating plate the sound wave is prevented from propagating due to the barrier the cavitation bubbles create. The density between water and metals is close enough that the transmission of sound waves between these two media is effective, which is why this cleaning technique is so useful.[10]

Cavitation Damage. As was stated above, the frequency of the sound wave determines the size of the cavitation bubble and the resultant implosion forces. The implosion energy at the lower frequencies is great enough to remove deposits that can include an oxide layer or any other thin passivation layer.[12] This can result in mechanical erosion of the surface being cleaned and is, of course, an important factor in the selection of a cleaning system.

Ultrasonic Frequency

Conventional ultrasonic cleaning tanks are available in discrete frequencies of 20, 25, 30, 40, 50, and 65 kHz. Some ultrasonic cleaning equipment is available in frequencies as high as 80 kHz to 1 MHz and more. One manufacturer is providing combinations of frequencies in the same cleaning tank.

Frequency vs. Cleaning Effectiveness. The knowledge that the lower the frequency the greater the implosion energy has been the basis for the selection of ultrasonic frequencies for cleaning systems through the decades. In 1959, Koontz and Amron published their work which showed that as the ultrasonic frequency decreased, the cleaning effectiveness increased. This work was based on particulate contamination using weighing and visual inspection techniques. In 1986, a study was reported by D. H. McQueen that evaluated cleaning effectiveness in terms of the type of contaminant to be removed. McQueen grouped contamination into two classifications: the microscopic or particulate contaminants which included particles from cutting, grinding, or polishing operations; and, the submicroscopic or molecular contaminants which included fats, oils, or proteins deposited as a very thin film such as from fingerprints or deposited from condensation of these soils. McQueen's study agreed with Koontz and Amron for particulate matter, the cleaning efficiency increased as the ultrasonic frequency decreased. However, for what McQueen has termed as molecular or submicroscopic contaminants such as fingerprints, the cleaning efficiency increased as the frequency increased. [5]

McQueen's hypothesis is that there is a different rate limiting mechanism for the two types of contaminants. The microscopic or particulate contamination is held to a surface by several bonds. In order to remove the particle, all the bonds must be broken at the same time; if they are not the few bonds that are broken are repaired by the time the next series of cavitation bubbles develop to break the remaining bonds. Therefore, the lower frequency, higher cavitation energy cleaning system is required to remove particulate contamination. McQueen states that the submicroscopic contamination such as fingerprints are held by a much smaller number of bonding sites. That in actuality the contaminant is removed at the molecular level, molecule by molecule, and is, therefore, dependent on diffusion as the rate limiting step. For this latter mechanism, the ultrasonic activity in the water increases the diffusion process and as the frequency increases the efficiency of the diffusion process increases. [5]

Ultrasonic Transducers

The transducers manufactured for cleaning equipment are available in two different types, magnetostrictive for 20 and 30 kHz and piezoelectric for 25 and 40 kHz and higher. The actual operating frequency of a transducer will vary ± 3 kHz from the design frequency.

Magnetostrictive Transducers. The magnetostrictive type of transducer is constructed of thin nickel strips placed together in a stack. A magnetic field is produced around this nickel stack through copper wire windings. The magnetic field causes the nickel atoms to align, taking up less space than the normal random atomic configuration. This phenomenon causes the nickel to contract. By causing the material to contract and relax, a resonant frequency in the nickel strip. The length of the nickel strip determines the frequency of the sound wave in the same manner as the length of an organ pipe, the longer the strip the lower the frequency. The length of the strips that are stacked together must be precision cut so that each individual strip resonates at the same frequency so the stack resonates in unison. This resonating frequency is then transmitted to the radiating plate and into the cleaning solution. [10] Magnetostrictive transducers can be made to produce frequencies greater than 20 or 30 kHz, however, the efficiency drops off dramatically as the frequency increases. [11]

Piezoelectric Transducers. The piezoelectric transducers used today for cleaning applications are made of lead zirconate-titanate ceramic crystals that are sandwiched together with electrodes that provide voltage to the crystals. There are two complementary effects that give rise to the phenomenon that is inherent in the piezoelectric crystal; when pressure (piezo- is taken from the Greek word piezein, to press)

is applied to the crystal, an electrical field is produced on the surface. Conversely, if an electrical field is applied to the surface, a strain is produced that causes the crystal to contract.[13,14] It is this latter condition that is utilized in the production of ultrasonic transducers. An alternating electric field is applied to the crystal at the desired frequency and causes it to contract and relax thus transforming the electrical frequency to the mechanical sound wave that is transmitted through the radiating plate and into the cleaning solution.

Transducer Bonding Techniques. Both types of transducers require bonding to a radiating plate, which is either the bottom of the cleaning tank or the face of an immersible transducer. There are two types of bonding techniques, brazing or epoxy bonding. The brazing technique produces a stronger bond and a better ultrasonic coupling medium; but, it requires a thicker radiating plate due to warping that occurs on a thinner plate with the braze heat cycle. Thus, brazing is limited to use with lower, higher energy frequencies, 20, 25, and 30 kHz. The epoxy adhesive bonding technique is used on thinner radiating plates and is typically used for the higher frequencies, 40 kHz and higher.

Power Requirements

The power levels in an ultrasonic tank system are a result of the amount of power the generator applies to the transducers and the number of transducers. The amount of power supplied to the transducers dictates the amplitude of the sound wave. The minimum power at which cavitation is produced, the cavitation threshold, is 0.3 Watts/cm² (1.9 Watts/in²) at the radiating plate.[12] The optimum power density for aqueous solutions was reported to be 2-3 Watts/cm² (13-19 Watts/in²) and for solvents at 1.5-2 Watts/cm² (10-13 Watts/in²) by Neppiras in a 1962 report.[11]

A power density measure that is more meaningful when discussing ultrasonic cleaning tank systems is the power per gallon of cleaning solution rather than the power per square inch of radiating plate surface. Cleaning systems are manufactured with varying power levels. Laboratory size tanks of up to about 10 gal generally have a power level of 100-200 Watts/gal. Newark Air Force Base uses small cylindrical 5-gal ultrasonic cleaning tanks that were designed to provide 500-600 Watts/gal.[3] Ultrasonic manufacturers, industry-wide, provide charts that show a significant decrease in power densities, 10-30 Watts/gal, as the size of the tank increases. However, this practice has not been supported by data that correlates cleanliness levels with the size of tank vs. power level.

The adjustment in power levels between small and large tanks is achieved by the number of transducers that are provided for the individual tank. The number of transducers directly influences the number of sites that will generate cavitation. Increasing the number of transducers improves the cavitation coverage and reduces the amount of "shadowing" to the different facets of a complex piece of hardware.[10]

TEST PROCEDURE

The specific details of the ultrasonic cleaning tank systems evaluated for this study are given in Table 1. These tank systems were used in various combinations in the three-step cleaning process. The frequency for cleaning steps 1 and 2 was held constant and the frequency for step 3 was either held the same or varied from steps 1 and 2.

Standard complex test specimens were contaminated and then cleaned as described below. The cleaning agents were Rocketdyne approved cleaners. After cleaning, the test specimens were evaluated for the remaining particulate matter and the nonvolatile residue (NVR).

Standard Complex Test Specimens. The test specimens were made of either 718 nickel base alloy or 304 stainless steel and were designed with through holes, blind passages, and threaded holes to simulate the complexity of the hardware, as shown in Fig. 1.

TABLE 1. Ultrasonic Tank Systems I.D. Code, Power Density, and Size

Tank System Frequency, kHz	I.D. Code	Volume Power Density, Watts/gal	Area Power Density, Watts/in ²	Dimensions I.D., inches	Soln. Cap., gal.
Laboratory Size Tank Systems:					
20	20-A	140	6	12x14x12 ht	7
20	20-B	100	10.5	10x19x16 ht	10
30	30-L	167	3.6	15.5xX9x6 ht	3
30	30-I	115	3.5	12x18x10 ht	6.5
40	40-B	210	6.2	9.75x14x10 ht	4
40+/-1	40-1	100	3.6	10x14x10 ht	5
40+/-2	40-2	100	3.6	10x14x10 ht	5
47	47	83	1.5	19.5x11x6 ht	4
Industrial Size Tank Systems:					
20	20-C	52	6.7	50x36x36 ht	230
25	25	25	4.6	48.5x54x31 ht	320
40	40-D	25	5.1	40x36.5x23 ht	145
50	50	25	4.6	48.5x54x31 ht	320
65	65	30	2.7	16x22x20 ht	32

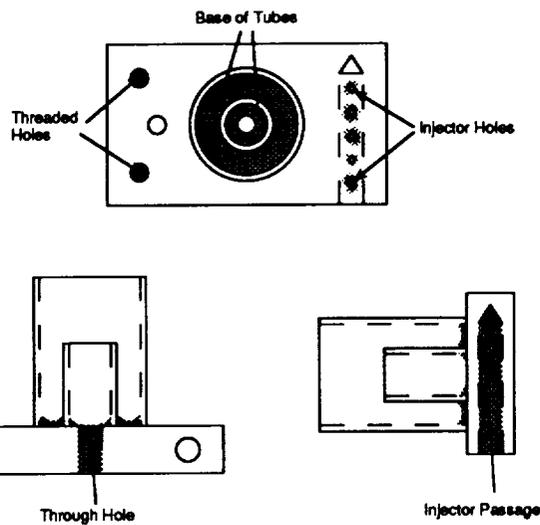


Fig. 1. Standard Complex Test Specimens with Placement of Contaminants.

Contamination of Test Specimens. The test specimens were contaminated, as shown in Fig. 1, with one of the following shop lubricants: 1) Cool Tool, by Monroe Fluid Technology; 2) Microfinish, by PetroChem Corp.; 3) Hydraulic Oil, by Mobil Oil Corp.; and 4) Centerpoint Lube, by Chicago Manufacturing and Distribution Co. Metallic fines were added to the contaminated areas and the specimens were heated in an oven at 200 F for 1 hr to simulate conditions of the machining processes.

Cleaning Procedure. The cleaning procedure was comprised of three steps. Each step consisted of placing the test specimens in the ultrasonic cleaning tank containing a cleaning agent, the ultrasonics were activated for 15 min, the specimens were removed, drained, immersion rinsed in hot deionized water, removed, and thoroughly flushed with ambient temperature deionized water. Each cleaning agent was diluted using deionized water. Cleaning Step 1 used a solution of 20% by volume of the emulsion degreaser Turco™ 3878 NC-LF*; Cleaning Step 2 used a 3.3 percent by weight (%/wt) solution of the mild alkaline cleaner Turco™ 4215 NC-LT plus 0.4%/wt solution of the nonionic surfactant Turco™ 4215 Additive; and Cleaning Step 3 used a 0.04%/wt solution of the nonionic surfactant Turco™ 4215 Additive. This last step was performed in a 100,000 Class cleanroom for the tests using the laboratory size tanks. The tests using industrial size tanks were conducted in the manufacturing shop which was not a controlled environment.

Laboratory Evaluation of Cleanliness. The cleanliness evaluation process was performed in a 30,000 Class cleanroom. Each specimen was evaluated by flushing with 500 ml of TCA. The TCA was filtered to extract the particles for particle count and the filtrate was evaporated to dryness and weighed to determine the nonvolatile residue. Particles greater than 400 microns were counted and recorded. The weight of the nonvolatile residue was divided by the surface area of the test specimen to calculate the amount of residue per surface area which was reported in mg/ft².

Comparison to Current Fine Clean Process Using TCA. The test specimens were contaminated and processed through the current manufacturing processes using aqueous solutions for preclean and vapor degreasing for fine clean. These specimens were then evaluated for cleanliness by the laboratory method given above.

RESULTS AND DISCUSSION

The cleaning effectiveness of each ultrasonic cleaning system was evaluated based on the nonvolatile residue and number of particulates remaining on the test specimens after cleaning. The NVR was further evaluated for the signal-to-noise ratio (S/N) which is a measure of the precision of the cleaning effectiveness. The calculation for the S/N was based on the Taguchi methods of analysis for results that aim for smallest-is-best.[15] The calculation used the following relationship:

$$S/N = -10 \log(\text{MSD}), \text{ and, } \text{MSD} = (y_1^2 + y_2^2 + \dots + y_n^2)/n$$

where MSD is the mean squared deviation, y is the NVR value in mg/ft², and n is the total number of data points for that test sequence.

Table 2 provides a list of the cleaning system combinations tested and the results. Fig. 2 provides a graph of the average NVR vs. the S/N, the data plotted also includes the comparison data for vapor degreasing. The best results on a graph for NVR vs. S/N for smallest-is-best are in the farthest lower right corner.

No obvious trend in effectiveness of frequency level or combination appeared on the graph of the overall results. Therefore, the data was grouped by manufacturer in order to compare ultrasonic frequency

*Trademark for Turco Products Division of Atochem North America, Inc., Westminster, California.

TABLE 2. Test Results of Ultrasonic Tank Systems
Listed in Order of Best Performance First

Cleaning Systems Steps 1&2/Step 3 by I.D. Codes	Tank Size Type	Avg. NVR, mg/ft ²	Signal-to-Noise Ratio(1)	Total Number Particles
20-B/20-B	Lab	1.7	-7.3	0
20-A/47	Lab	2.7	-8.7	0
25-D/40-D	Indust.	3.0	-9.8	0
40-1/40-2	Lab	3.0	-10.5	9
25-D/25-D	Indust.	3.2	-11.3	0
25-D/50-D	Indust.	3.6	-12.3	1
47/47	Lab	3.9	-13.4	0
20-C/20-C	Indust.	4.1	-13.8	0
20-C/65	Indust.	4.8	-13.7	3
30-I/47	Lab	4.9	-14.1	0
40-B/40-B	Lab	5.0	-14.5	0
20-B/40-B(2)	Lab	5.3	-15.0	4
30-I/30-I	Lab	5.6	-15.2	0
20-B/40-B(3)	Lab	5.7	-16.4	0
30-L/30-L	Lab	5.8	-16.5	0
40-1/40-1	Lab	5.8	-17.2	9
40-B/20-B	Lab	6.1	-16.8	5
47/20-A	Lab	10.7	-20.6	0

- (1) Signal-to-Noise Ratio = $-10 \log(\text{Mean Squared Deviation})$.
 (2) Soil remained on these test specimens for 2 days.
 (3) Soil remained on these test specimens for 24 days.

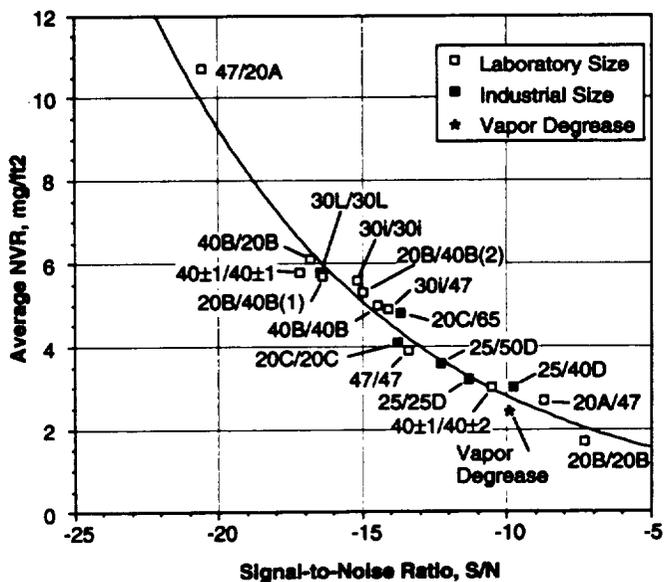


Fig. 2. A plot of the results of the cleaning effectiveness of the ultrasonic systems evaluated.

combinations while holding design differences a constant, Fig. 3a-f.

There were several ultrasonic cleaning systems that provided cleaning effectiveness close to that of vapor degreasing as can be seen in Fig. 2.

The best overall test result was for the cleaning sequence 20B/20B which appears to be a possible anomaly, Fig. 3a. It would be expected that the 20B/40B results would at least be somewhere between 20B/20B and 40B/40B, if frequency combination order were not an influencing factor. Therefore, the test for 20B/40B was repeated, but similar results were obtained. This would indicate that the results for either 20B/20B or 40B/40B were not accurate.

The results of three of the cleaning sequences, Fig. 3b, c, and d, appeared to support the suggestion that a low frequency to remove the greater deposits of soil followed by a high frequency to remove the last microlayer of soil enhances cleaning effectiveness. In Fig. 3b, results are shown that indicate that frequency combination order are an influencing factor. The cleaning sequence results provided in Fig. 3c indicate that cleaning with a higher ± 2 sweep frequency in the last cleaning step improves the cleaning effectiveness over that of using the lower ± 1 sweep frequency in all three cleaning steps. The cleaning sequence results provided in Fig. 3d show that cleaning with 25 kHz followed by 40 kHz in the last cleaning step gives improved cleaning effectiveness over using 25 kHz for all three steps. The system using 25 kHz followed by 50 kHz was not very effective. The 50 kHz ultrasonic system did not perform well because this system used 25 kHz transducers that were driven at 50 kHz by adjusting the generator. To drive the ultrasonic transducers at a frequency different from the design frequency does not appear to provide an efficient transfer of energy and as a result its performance is poor. This same type of situation was also evaluated by A. A. Busnaina, et al., and reported to be ineffective.[16]

It was visually observed that if a water break free surface was not obtained after Step 2, Step 3 was not very effective. With the 20C/20C and 20C/65 systems, very poor results were being obtained after Step 2 with the 20 kHz system, Step 3 added significantly to these cleaning processes. As can be seen in Fig. 3e, no significant difference in cleaning effectiveness was observed between these two different processes.

The ultrasonic frequencies of the cleaning systems were classified as a low frequency system for 20, 25, and 30 kHz and as a high frequency system for 40, 50, and 65 kHz. Study performed by D. H. McQueen used much higher frequencies for the high frequency evaluation. The 50 and 65 kHz systems tested in this study were still being considered as experimental equipment. Therefore, the high frequencies McQueen used are not currently available in industrial size equipment.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of this study, it has been concluded that a cleaning system should be tested to evaluate its effectiveness on the specific substrate and soil that must be cleaned. The overall performance of the frequency levels, low or high, varied from manufacturer to manufacturer. It appeared that the ultrasonic generator-transducer-radiating plate design differences between manufacturers were more of an overriding factor in cleaning effectiveness than the frequency levels or combinations of frequencies in the cleaning system. However, based on the limited tests performed, it appeared that, when the manufacturing design of the system was held constant, the use of low frequency ultrasonic systems in preliminary cleaning steps to remove gross amounts of soil followed by high frequency to remove the microlayer of soil in the final cleaning step gave enhanced cleaning effectiveness. It was also determined that lower power levels are acceptable for industrial size tanks compared to the smaller laboratory size tanks, but a little higher than that recommended by the manufacturer, at least for the Rocketdyne application.

The system selected by Rocketdyne was 25 kHz for cleaning steps 1 and 2 and 40 kHz for step 3. The power levels requested were 4.2 watts/in² at the radiating plate with 43 watts/gal for each tank.

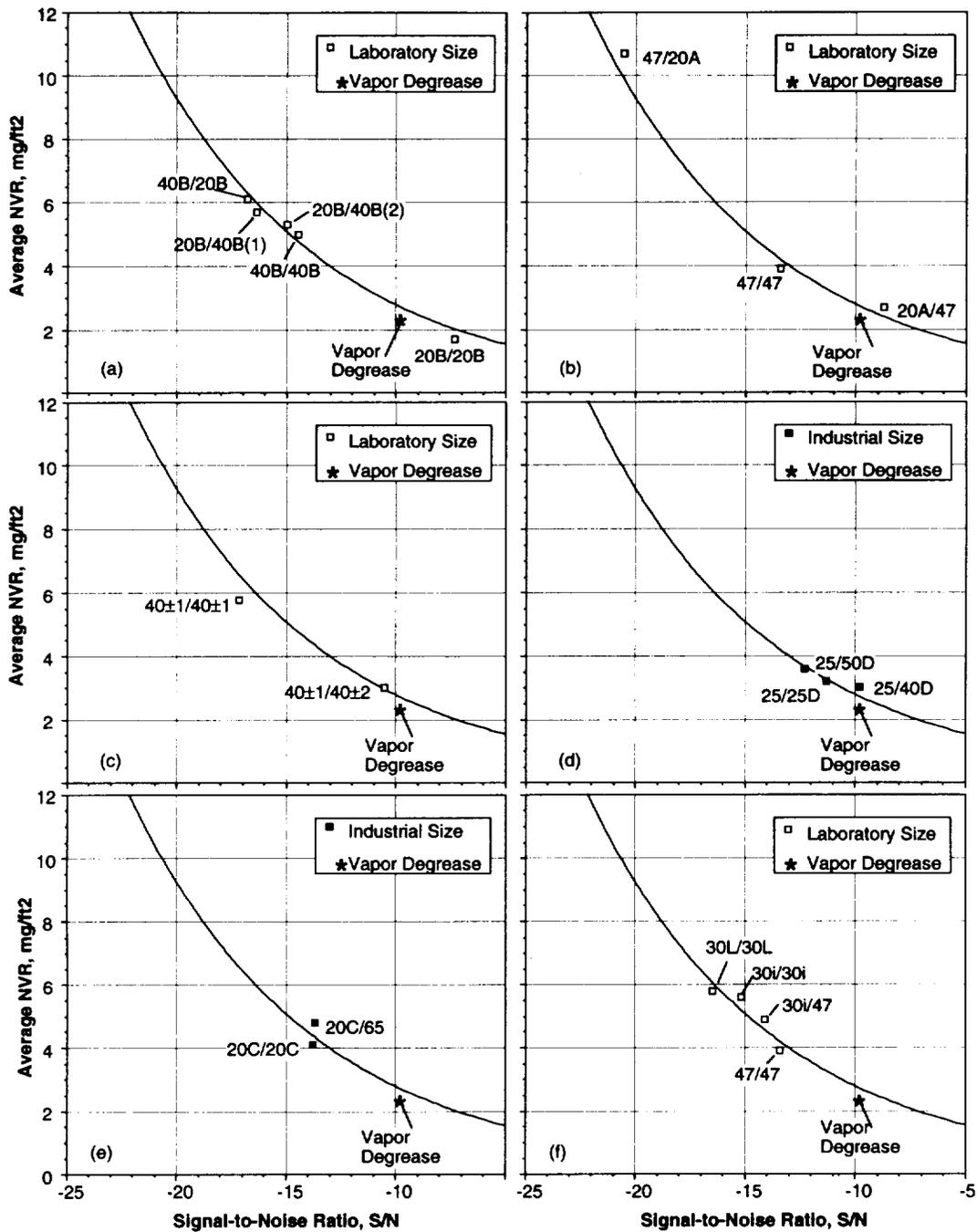


Fig. 3. A plot of the cleaning effectiveness of the ultrasonic systems by manufacturer.

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